Project ID: MAT165_grant_2019

Pacific Northwest NATIONAL LABORATORY

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Direct-Extruded High-Conductivity Copper for Electric Machines

(Agreement ID 69145)

GLENN J GRANT

XIAO LI, KEERTI KAPPAGANTULA

PACIFIC NORTHWEST NATIONAL LABORATORY

JAMES SCHROTH, HONGLIANG WANG, CHEN ZHOU

GENERAL MOTORS RESEARCH AND DEVELOPMENT LABORATORY

2019 DOE VEHICLE TECHNOLOGIES PROGRAM

ANNUAL MERIT REVIEW AND PEER EVALUATION MEETING

JUNE 12, 2019

WASHINGTON, DC

Overview

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Timeline

- Start date: Sept 2017
- Project end date: Dec 2019
- Percent complete: 70%

Budget

- Total project funding: $1.15M
  - DOE: $600,000
  - Industrial cost share: $550,000
- Future Funds Anticipated: $ 0

Barriers

- Need Increased Efficiency – bring power density above 5 kW/L
- "Reduction in the volume of the components is necessary to enable electric traction drive systems to fit within the increasingly smaller spaces available on the vehicle. Motor volume reduction is limited by the flux density capacities of materials used in current electric steels and electrical conductivity limitations of copper windings"[1]
- Need higher conductivity electrical conductors

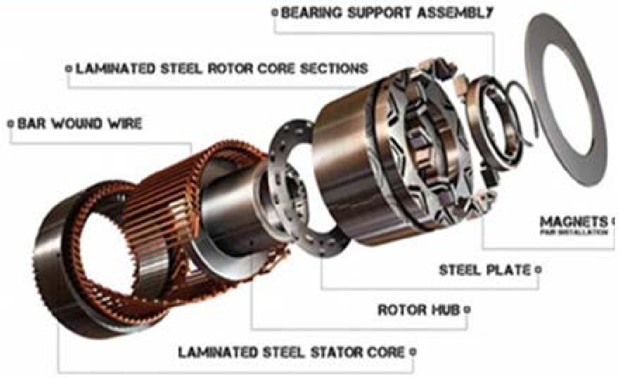
Partners

- CRADA with General Motors Research and Development
- Project lead: PNNL

[1] U.S. DRIVE Electrical and Electronics Technical Team Roadmap October 2017
https://www.energy.gov/eere/vehicles/us-drive-partnership-plan-roadmaps-and-accomplishments

Relevance - Impact

Permanent Magnet Motors



Induction Motors

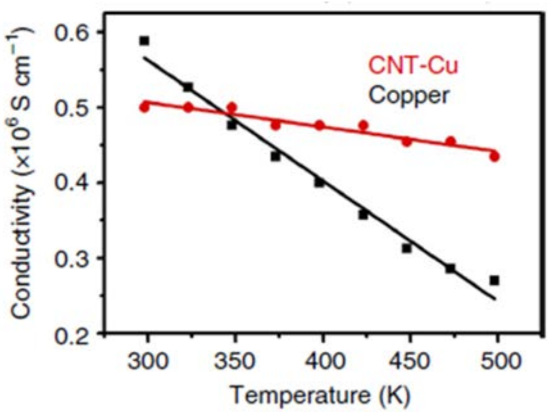
- The efficiency, weight, power density, and therefore the cost of an electric machine could be improved if the primary conductors could be made with much higher conductivity than standard electric copper.

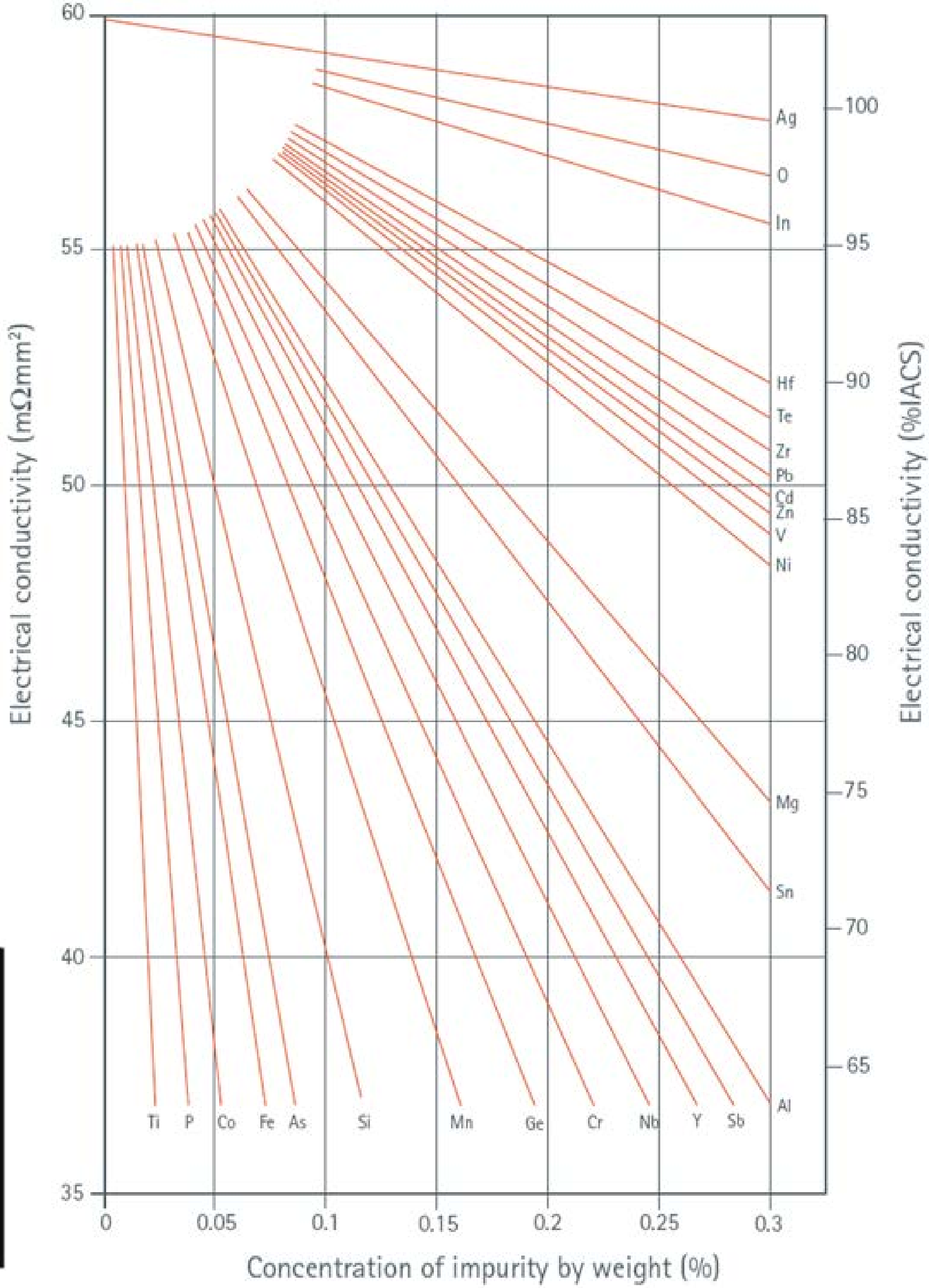
Relevance

The challenge of increased conductivity:
– pure copper is already very conductive

- Anything added to it makes it worse
- In addition, as temperature increases in electric motors, electrical conductivity of the pure copper components goes down, reducing motor efficiency

…With the possible exception of Carbon





Relevance

Carbon - Copper Composites

- Fully dense Cu-C Composites have been made that show conductivity better than pure copper at elevated temperature
- Carbon is immiscible with copper so the effort is to see what kind of process can be used to make the composite and..
- if it actually increases conductivity

Forms of Carbon

- Graphite
- Reduced Graphene Oxide
- Graphene
- Single and Double Wall Carbon Nanotubes
  -metallic or covalent

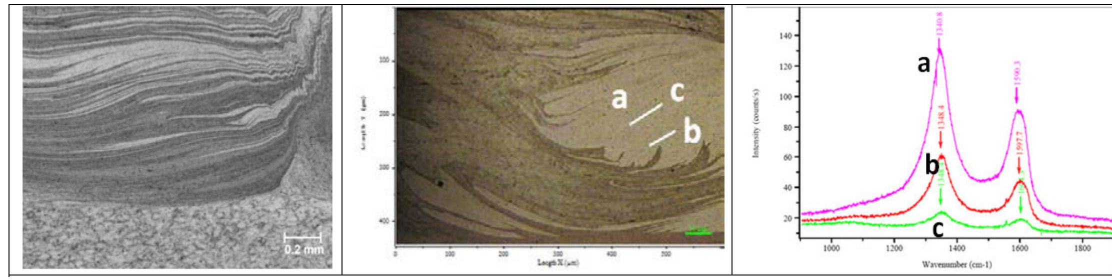
Possible manufacturing Process

- Layered Structures (PVD-CVD- other thin film methods)
- Super saturated solutions from ball milling or Magnetron Sputtering
- Bulk Composites from melt solidification Processes
- Covetic processing (melt solidification in a magnetic field?)
- Hot Extrusion

To date, few bulk methods have been successful at demonstrating increased conductivity

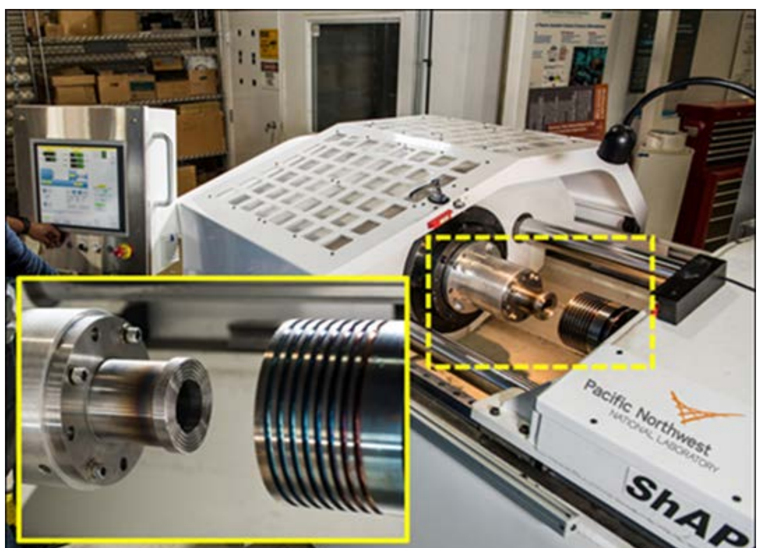
Relevance – Need a new bulk manufacturing process to form the Copper-Carbon Composites

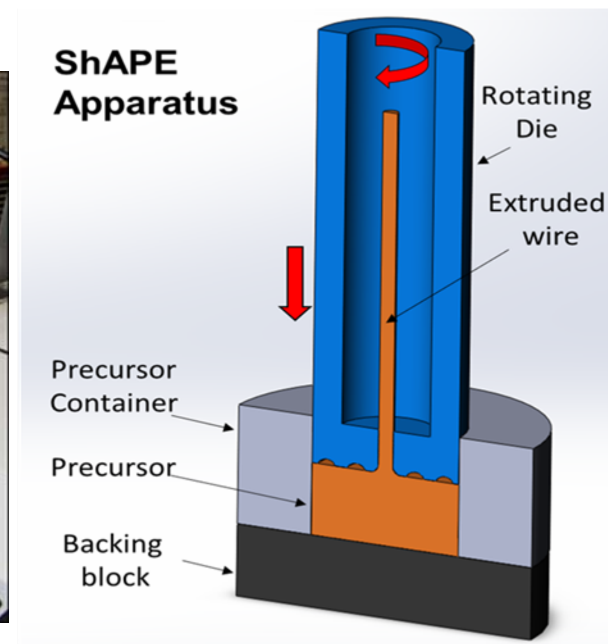
We have observed that super-saturated and non-equilibrium mixtures and alloys can be made using severe plastic deformation



We have developed techniques at PNNL to combine this severe plastic deformation process with an extrusion process to make it scalable

Shear Assisted Processing and Extrusion (ShAPE)





Relevance - Approach

➢ Objectives

- Develop and demonstrate a high conductivity copper-based material composed of a copper-carbon composite.
- The copper composite will be fabricated using the ShAPE process
- Show that ShAPE can shear, mix to a high level of homogenaity, and extrude wire and bar that is a combination of copper and various forms of carbon (including graphene and graphite)

At the conclusion of the project the composite will be formed into wire and shorting bar product forms and assembled into an induction motor rotor demonstrator for testing.

Relevance - Approach

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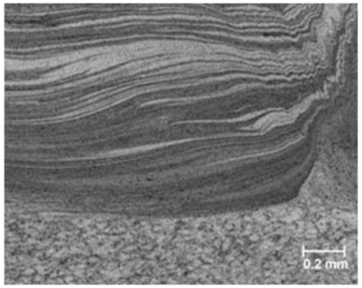
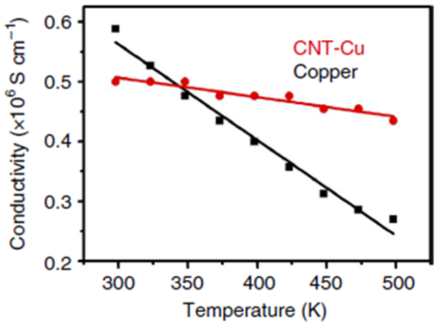
- Task 1 Making a good composite
  - Methods to mix carbon (Graphite and Graphine) into copper
  - Die fabrication and testing
  - Extrusion Trials
- GATE - Electrical resistivity at temperature
- Task 2 Making the correct shape extrusion
  - Rod, strip or thin plate, shape?
- Task 3 Joining of wire/strip
- Task 3 Roll forming the shorting bar or square section(?) wire)
  - Ductility issues?
- Task 4 Rotor assembly by FSW
- Task 5 Rotor testing (electrical continuity and resistivity at elevated temp)





Milestones

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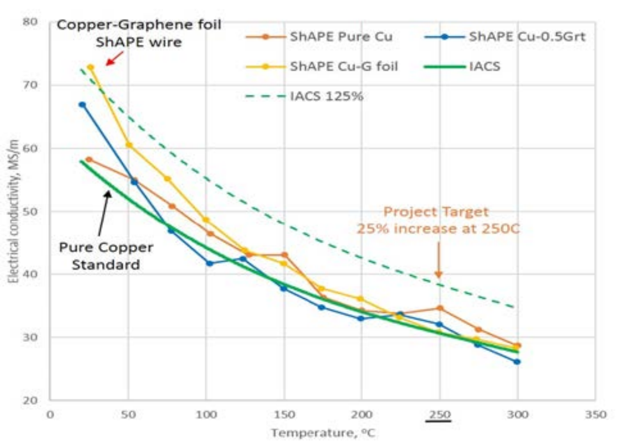
Milestone 1	Micrographs showing composite cross section free of defects in the composite	Completed
Milestone 2	At least one of the copper carbon composites must show electrical performance (elevated temperature conductivity) significantly higher than current pure copper materials. The degree of improvement metric will be determined from motor efficiency analysis or by discussion with GM engineering, but the metric is anticipated to be at least 25% higher conductivity at elevated temperature (250C)	Completed*
Milestone 3	Batch to batch electrical conductivity does not show significant variability	Complete
Milestone 4	Copper composite weld joint on rod material with joint strength at least 80% of base material and small to insignificant electrical losses across joint	Complete



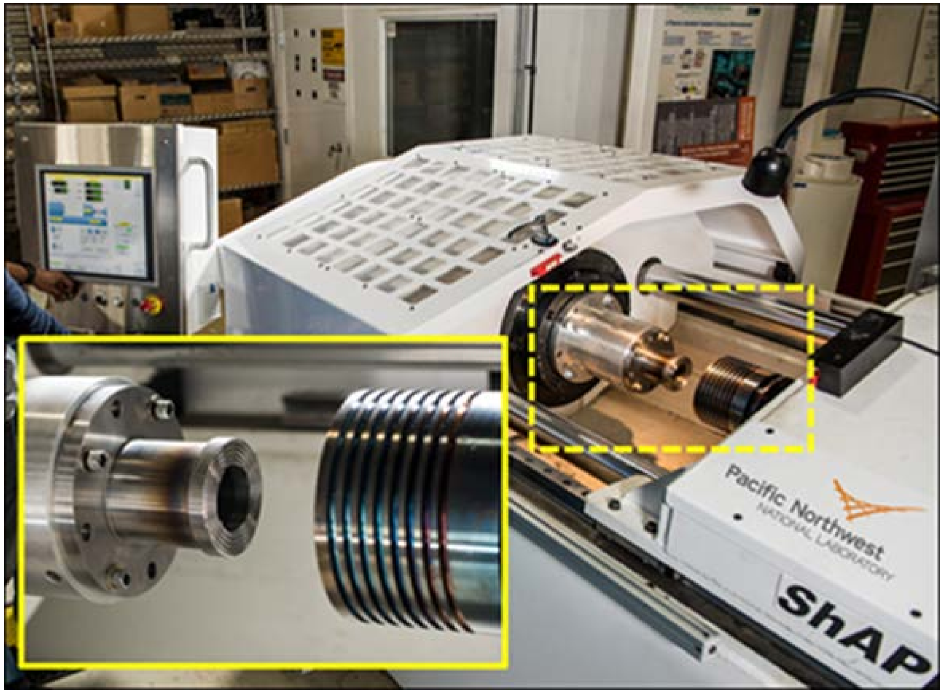
Experimental setup and materials

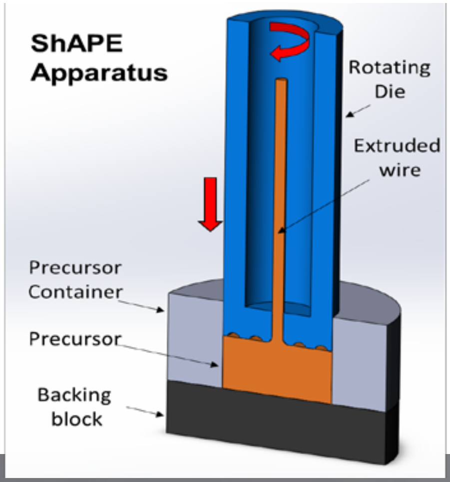
Precursor material:

Copper powder (<45um, 99.7% purity), blended with 1%-15wt% Graphite powder (<45um, >99.9%)

OR

Solid Copper cylinder (C10100, <99.99% purity), Drill and filled with Graphite powder (<45um, >99.9%)





1”OD Scroll face ShAPE die with 0.1” central orifice.
Extrusion ratio = 100:1

Thermocouple inserted to 0.5mm to die face and connect to a wireless data transmitter.

Carbon content ranges from 0.3wt% to 15wt%

Technical Accomplishments and Progress: Task 1 Process Development

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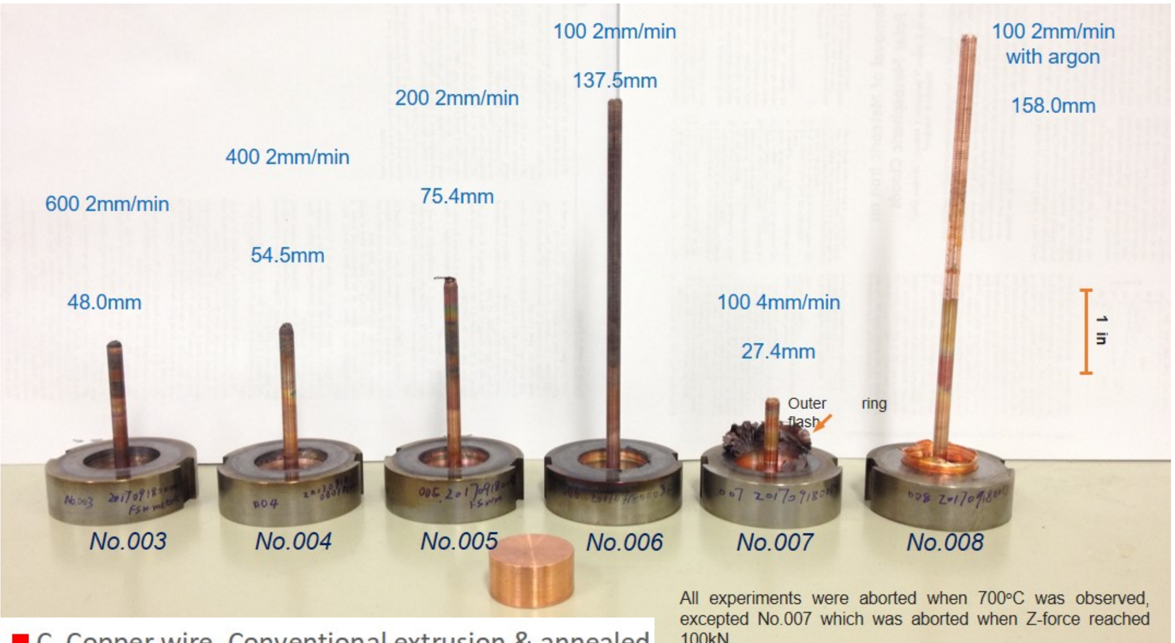
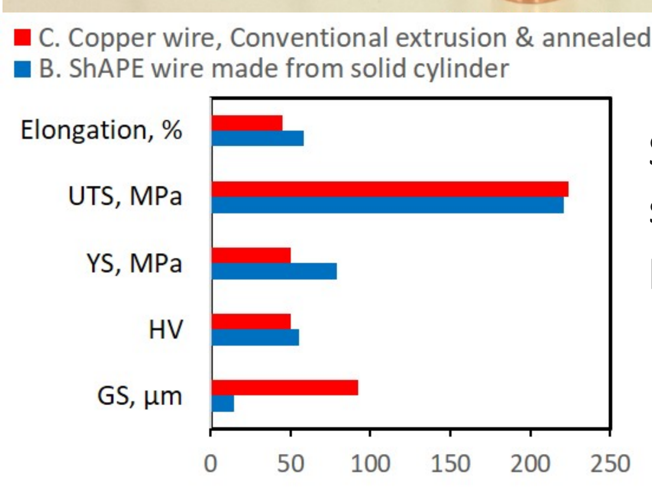
Stage 1: Copper wire extruded from solid copper disc

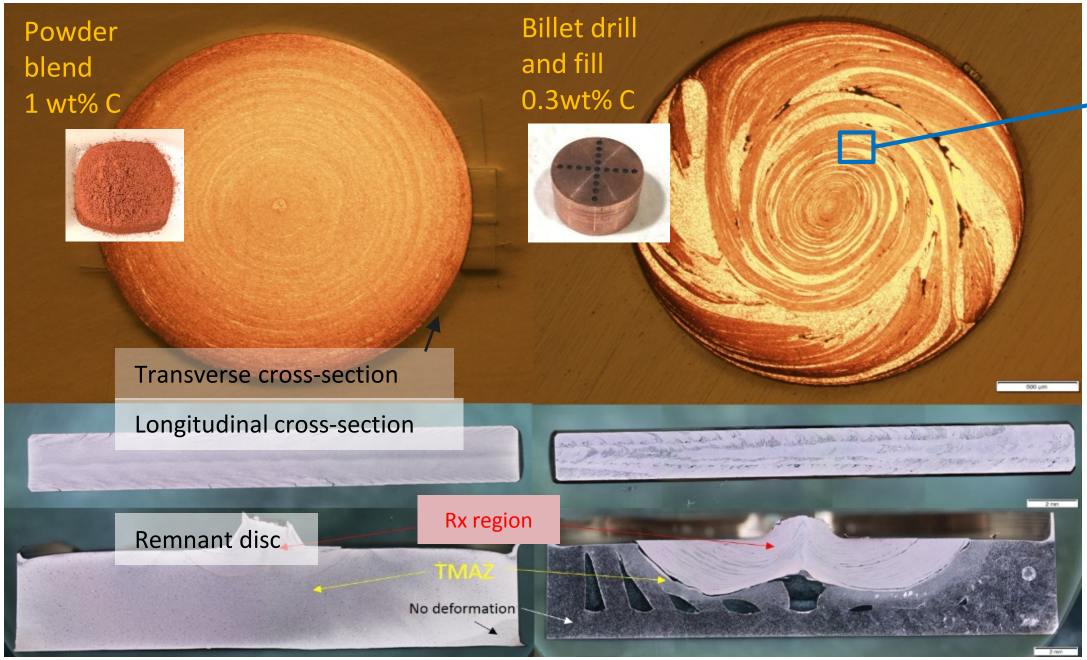


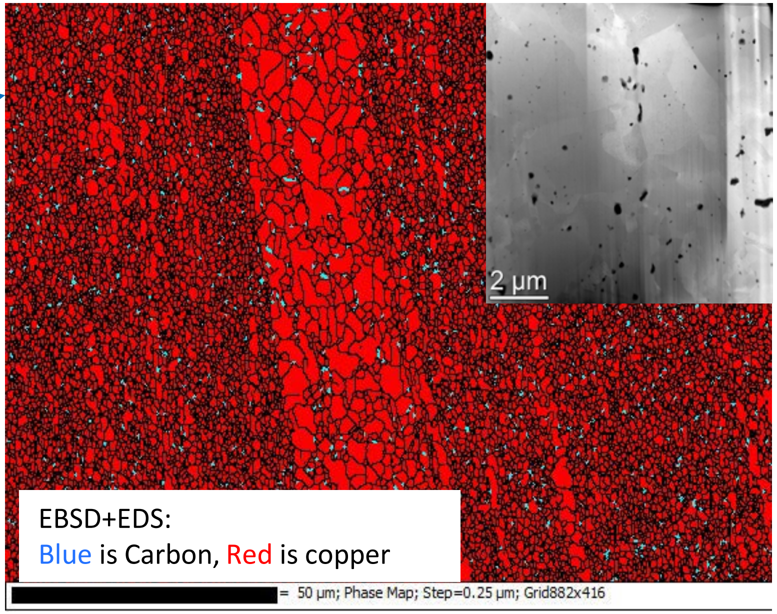


ShAPE extruded wire shows good mechanical properties

Process development during FY18-19 has led to a consistent process capable of producing continuous wire extrusions over 600mm in length (limited by can or billet volume)

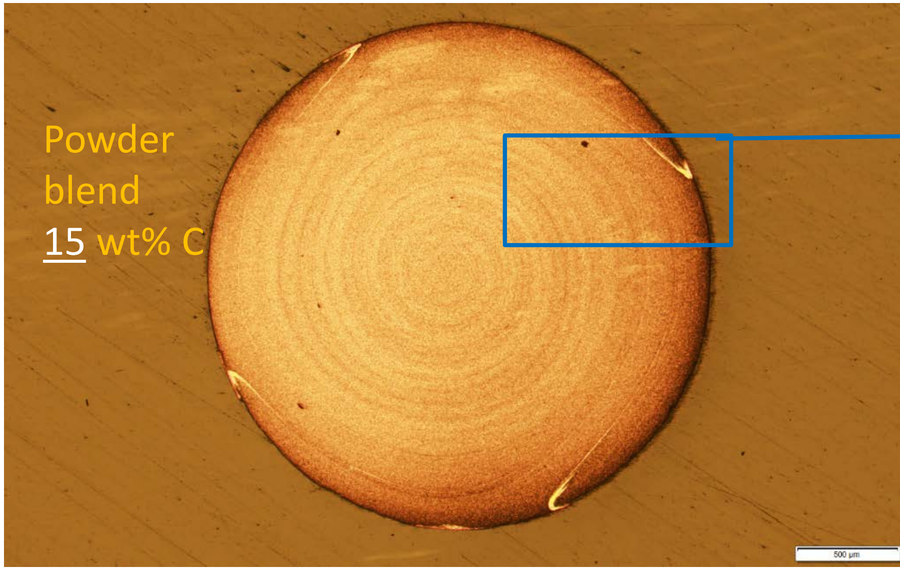
Technical Accomplishments and Progress: Task 1 Process Development

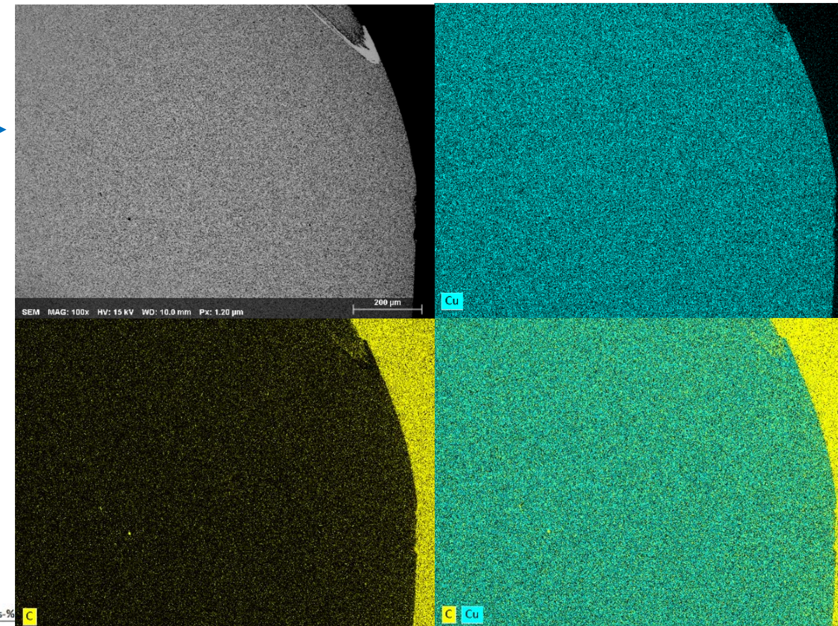


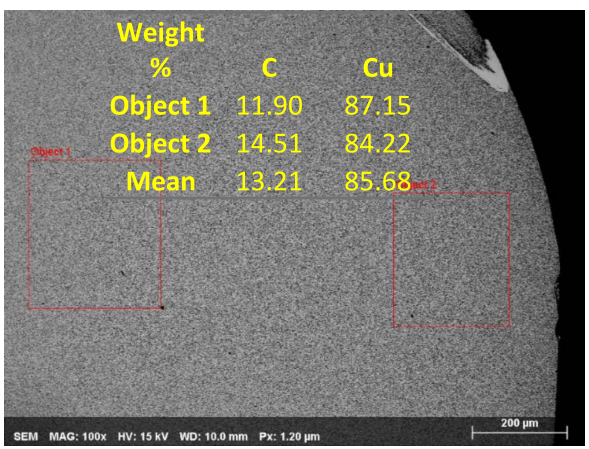


- **Carbon was uniformly dispersed in the copper matrix, even the in drill-and-fill sample.**
- **Size of carbon particles has been refined from ~30um to <1 um or even nano-size.**

Technical Accomplishments and Progress: Task 1 Process Development

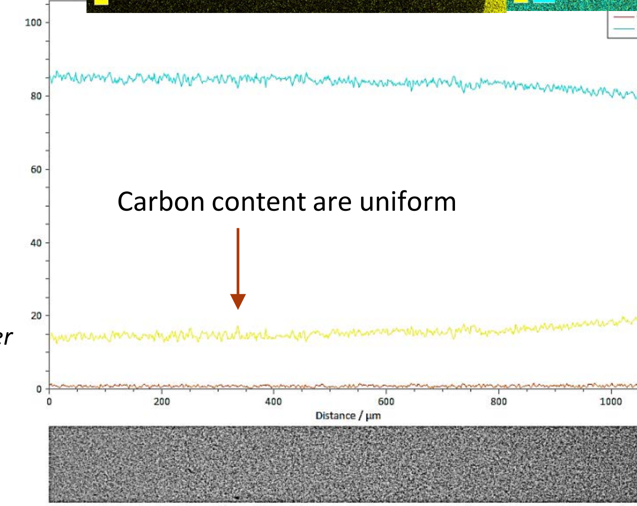






Weight %	C	Cu
Object 1	11.90	87.15
Object 2	14.51	84.22
Mean	13.21	85.68

Center



Edge

Technical Accomplishments and Progress: -Wire Texture

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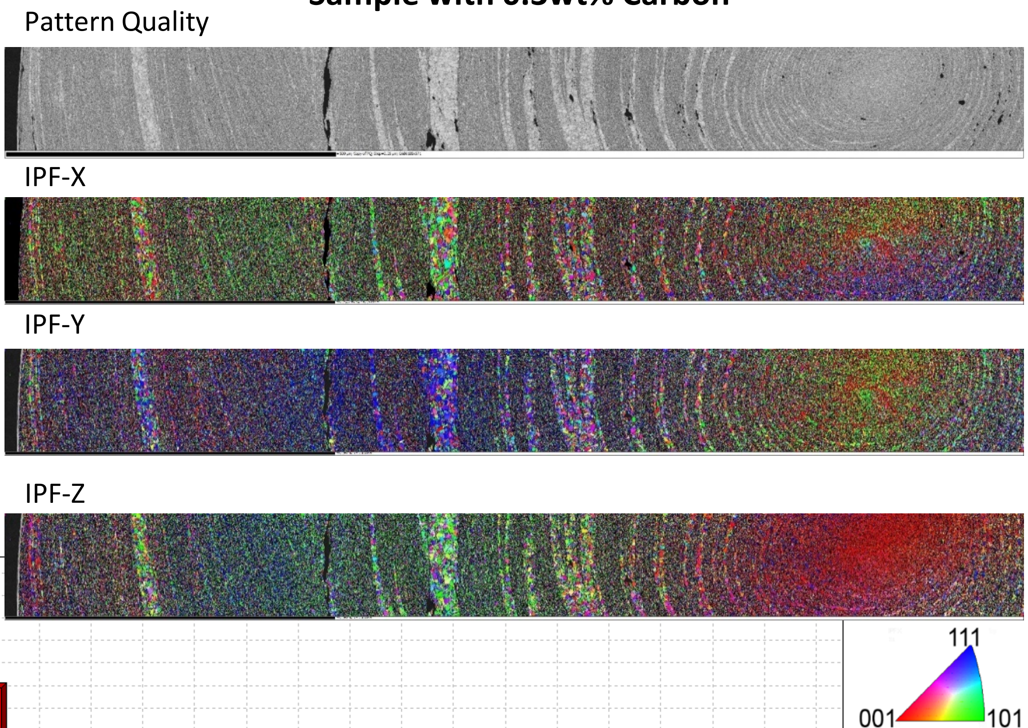
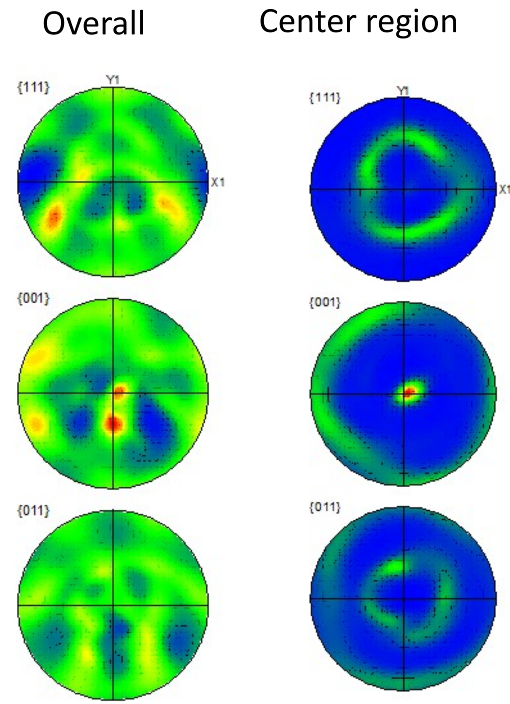
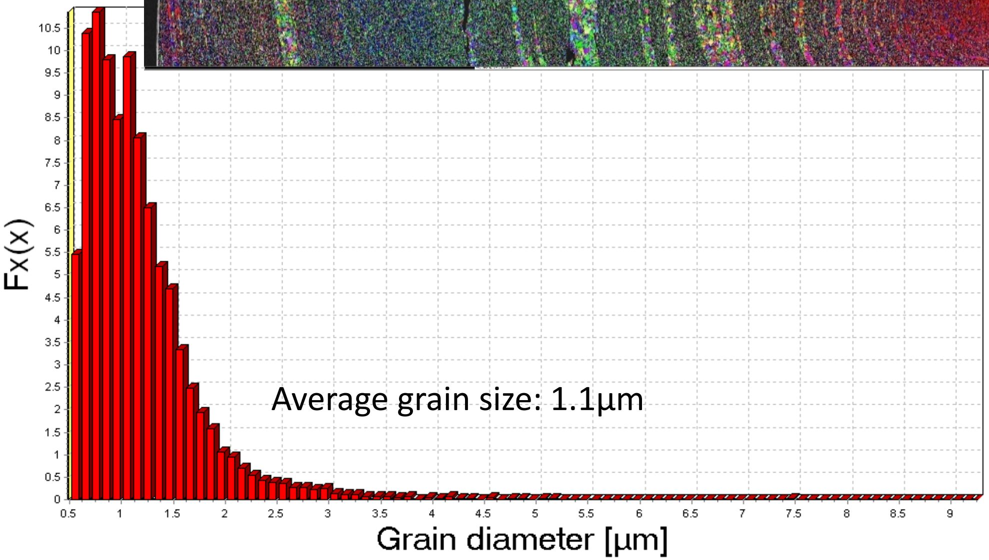
Sample with 0.3wt% Carbon







- Graphite particles impede grain growth and refined GS from 78 to 1.1µm.
- Wire has isotropic texture, except the center shows {001} texture.

Technical Accomplishments and Progress: Electrical Testing

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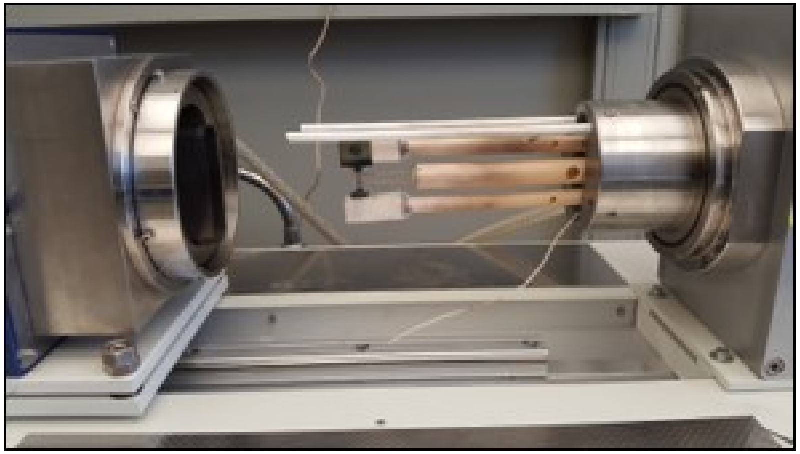
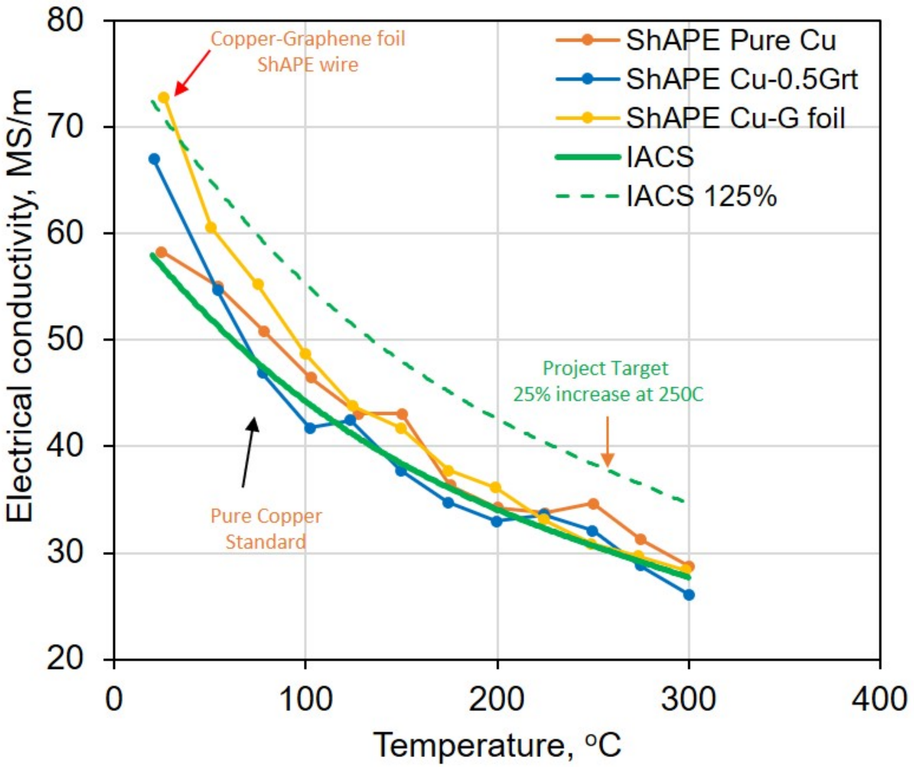


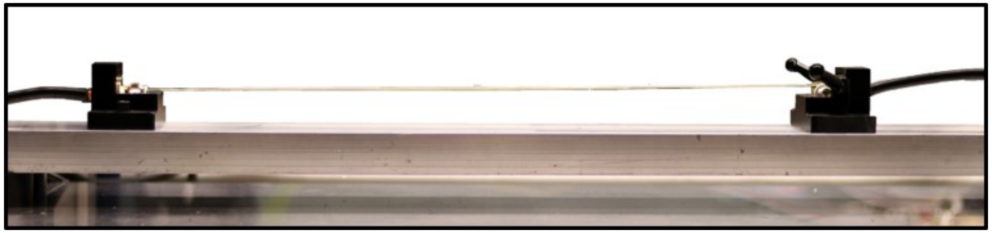
The electrical conductivity from room temperature to 250^{o}C was measured using 4-probe method in a furnace with Argon protection gas.

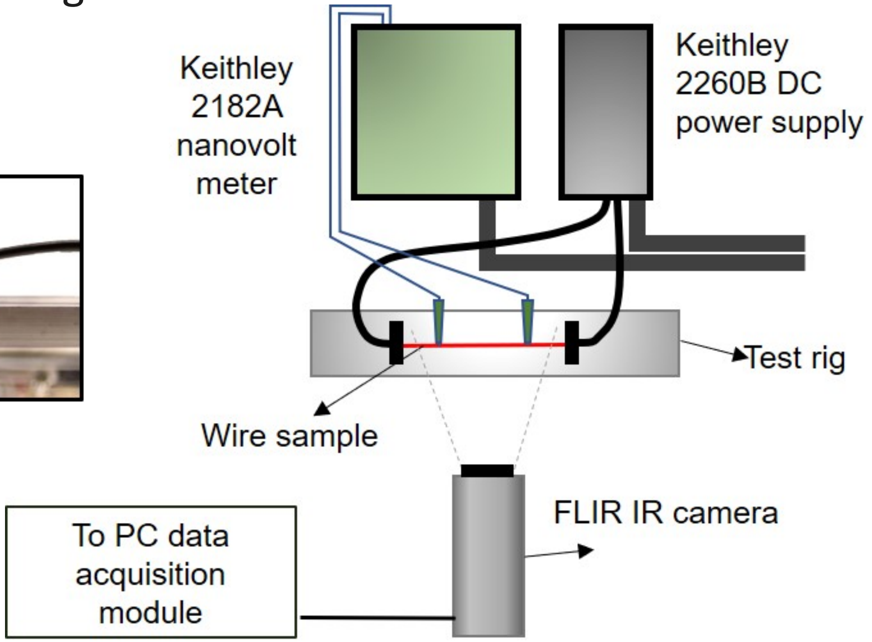


- ➢ The electrical conductivity of pure copper ShAPE wire is the same or slightly better when compared to pure copper.
- ➢ Copper-**graphite** ShAPE wire is similar in electrical conductivity to pure copper
- ➢ Copper-graphene ShAPE wire is 25% better than pure copper at room temperature but drops at elevated temps.
- ➢ We are skeptical of these results
  - ➢ Error bars on this method are very large, and the sample measured length is small

Technical Accomplishments and Progress: Electrical Property Measurement Performed at Ohio University

Conductivity measurements done by Keerti Kappagantula and her students at Ohio University gave us much higher fidelity conductivity measurements



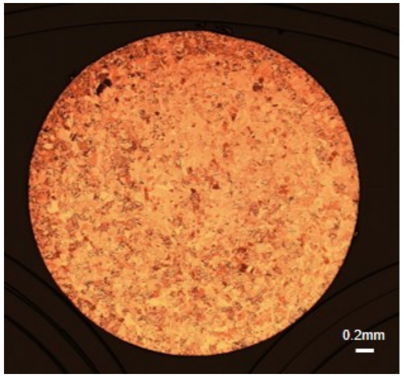


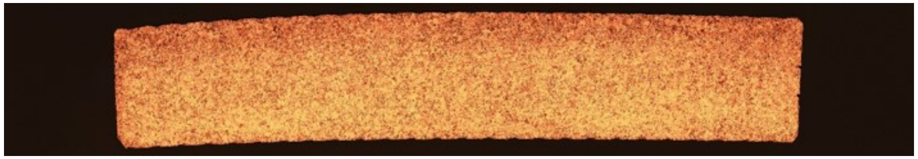
Custom designed electrical performance measurement set-up using non-contact infrared thermometry, nano-voltmeter and high-current power supply. Electrical conductivity and current density at elevated temperatures are measured using this set-up.

Technical Accomplishments and Progress: Electrical Property Measurement Performed at Ohio University

Copper wires from solid copper disc and graphene

- Two types of graphene were used:
  - Reduced graphene oxide powders
  - High-purity monolayer graphene sheets





Property	Control sample	Copper/ monolayer graphene
Electrical conductivity at 20°C (%IACS)	100.75 ± 0.002	103.57 ± 0.001 (**2.8%** ↑)
Electrical conductivity at 60°C (%IACS)	87.27 ± 0.0146	88.43 ± 0.0296 (**1.3%** ↑)
Electrical conductivity at 90°C (%IACS)	79.57 ± 0.0146	80.23 ± 0.0296 (**0.8%** ↑)
Temperature co-efficient of resistivity (/°C)	0.0037	0.004

Responses to Previous Year's Reviewers' Comments

- This is the first year that the project has been reviewed

Collaboration and Coordination with Other Institutions

Technology Transfer / Collaborations

Shape Wire process development, tool design, and coupon testing
PNNL

Testing for Mechanical and Electrical Performance
PNNL
GM
OU

Welding and joining trials on copper composite wire
GM

Motor component testing
GM
Electrical efficiency testing

Commercial Component Development
GM

Collaboration

- Biweekly conference calls where laboratory work scope and tasks are discussed
- GM provides electrical testing.
- PNNL executes FSW process development and tool design
- GM executes welding and joining trials on copper composite wire
- GM provides all component scale testing including electrical efficiency tests
- Over the 3 year project GM will participate at approximately 50% of the total budget through in-kind contribution

Performance data and manufacturing technology will be transferred to industry through the mechanism of a Cooperative Research and Development Agreement (CRADA) with General Motors (GM), ensuring a clear path to commercialization.

Remaining Challenges and Barriers

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- Can higher Graphene loading result in higher conductivity
- TCR should go other way. Need to discover why we are not seeing a well known effect that certain carbon (covalent) compounds show, increased conductivity with increased temperature
- We also need to adapt the process to produce a Cu-composite shorting bar shape for insertion into induction motor rotor.
  - Graphite/Graphene levels are so low, we have seen the ductility to be similar to pure copper

Proposed Future Work

- Manufacture copper composite wires with lower defect density graphene

6" X 6" Monolayer graphene on Copper foil

Cut into ½" X ½" flakes

Load the flakes into a solid cylinder with a square hole

- Roll copper composite wires into shorting bar cross section, assemble induction rotor and friction stir weld to complete assembly

<u>Resistance test</u> - The resistance for the rotor across end cap and 180 apart is good. Same as cast copper rotor

<u>Electrical continuity and homogeneity of welds</u> - Induction test identifies quality of bar to end cap weld. All welds are the same and similar to the cast rotor performance.

Any proposed future work is subject to change based on funding levels

Summary

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Accomplishments

- ✓ We have used Shear-assisted extrusion and processing (ShAPE) to fabricate Copper-carbon composite wire with 0.1” diameter
- ✓ We have developed extrusion tools and dies for producing Copper-carbon composites .
- ✓ We are developing fully dense extrudates by conducting parametric studies of the process parameters

Technical highlights

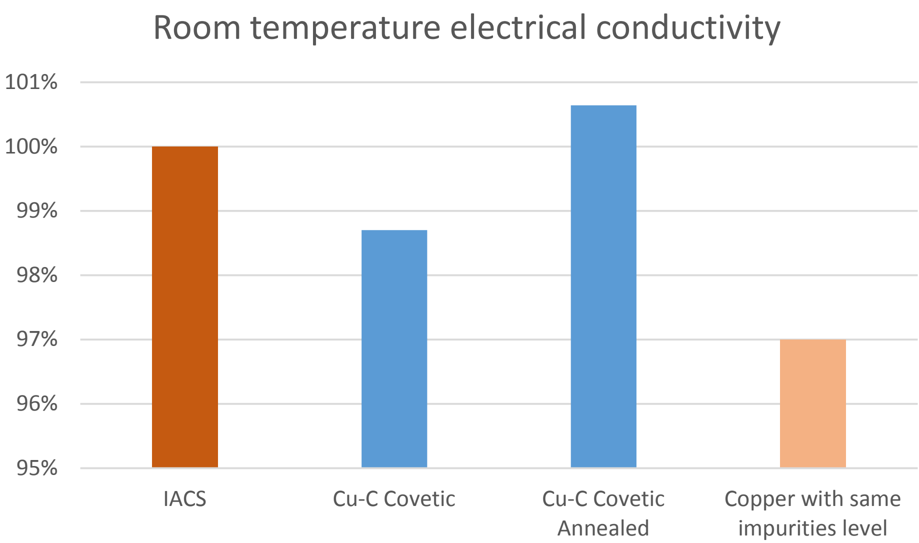
- ✓ Mixed and densified copper and carbon powder to a solid puck and extruded copper-carbon composite wire with good integrity and properties
- ✓ We have seen a verifiable increase in conductivity as high as 3% over ICAS Copper
  - ✓ Wire manufacturers we have consulted consider this a significant increase

Impact toward VTO Objectives

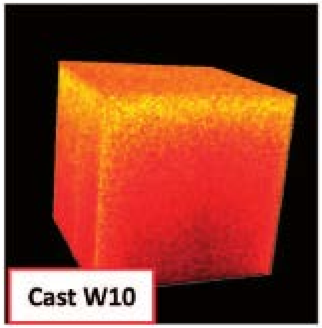
This project will leverage a new manufacturing process to manufacture high conductivity wire by an unconventional method involving high shear and plastic deformation. It could be disruptive to the way automotive traction drives are constructed, sized or powered. The high conductivity wire for stator winding could spill over into permanent magnet motor space where there are also strong incentives to improve motor performance.

Technical Backup Slides

- 1. Covetic Carbon-Copper (2 to 5 wt%)



Secondary Ion Mass Spectrometry (SIMS) shows carbon presence in the copper lattice



Theory:

New material

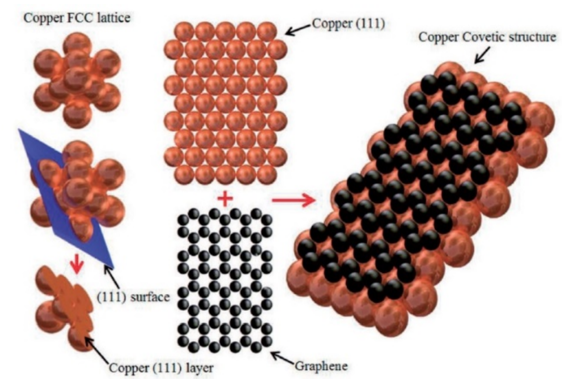


Fig. 1. Idea of copper Covetic material nanoscale structure

Barriers:

1. A significant improvement is seen in Al Covetic, but not in Cu
2. This result is not obtained by all researchers

ShAPE:

Can ShAPE to make this unique structure from graphite?

* ”Fabrication and Cold Drawing of Copper Covetic Nanostructured Carbon Composites”

Theories and barriers

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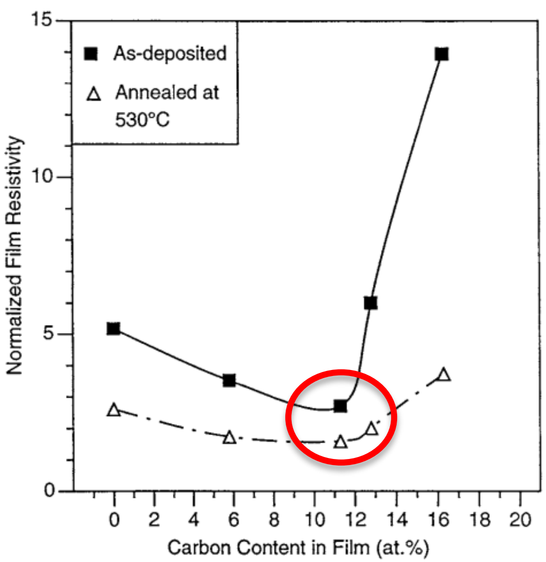
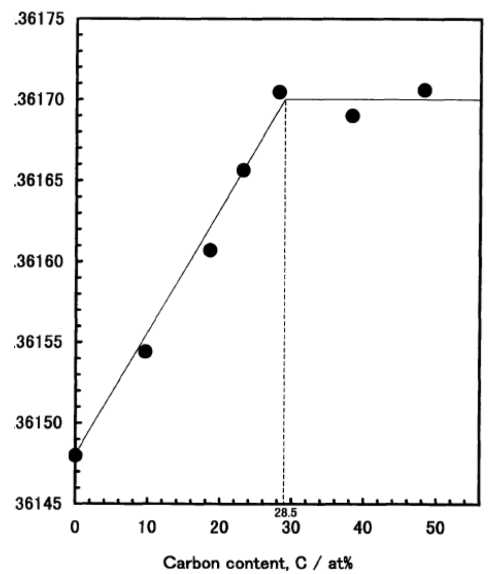
2. Copper Supersaturated solid solution

Non-equilibrium super saturated solid solution made by high energy mechanical alloying or magnetron sputtering deposition. Reduced resistivity with increased carbon content up to 12 at% (2.5 wt%)*





Theory:

Non-equilibrium super saturated solid solution increased lattice parameter which increased electron free path.

New material

Carbon form does not matter.

Barriers:

1. Only few papers reported this in the last 20 years.

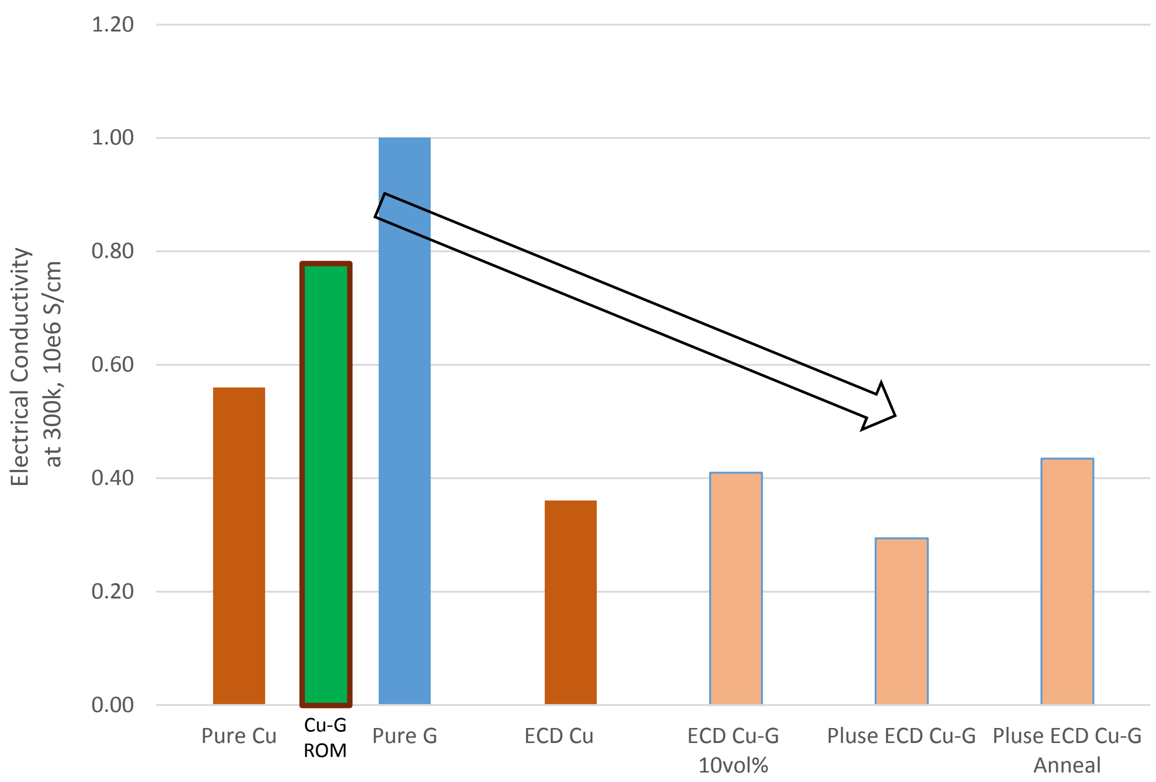
ShAPE:

Can ShAPE make this unique structure from graphite?

*Chu, J. P., et al. "Microstructure and properties of Cu-C pseudoalloy films prepared by sputter deposition." *Metallurgical and Materials Transactions A* 29.2 (1998): 647-658.

**Saji, S., et al. "Solid solubility of carbon in copper during mechanical alloying." Materials Transactions, JIM 39.7 (1998): 778-781.

3. Cu-G (Graphene)



Theory:

Graphene (G) has better electrical conductivity than copper.
<u>Rule of mixtures approach</u>

Barriers:

1. High quality, large area Graphene is desired. Thus the cost increases.
2. Effect only seen at lab scale and difficult to scale up.
3. Voids introduced during synthesizing composite make it eventually worse than pure Cu.

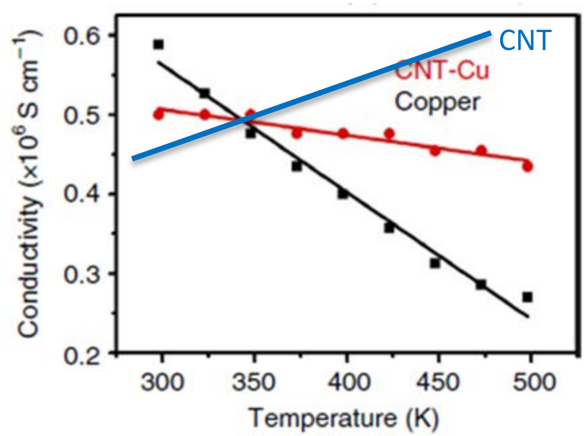
ShAPE:

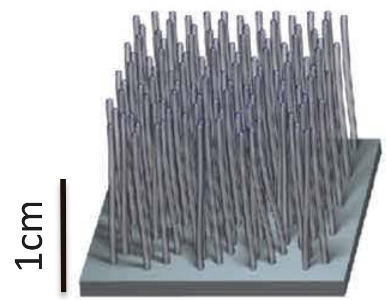
1. *Can ShAPE make G from graphite by high shear?*
2. *Can ShAPE make voids-free Cu-G composite at bulk scale*

a. Electrical conductivity of copper–graphene composite films synthesizedby electrochemical deposition with exfoliated graphene platelets
b. A New Electrochemical Approach for theSynthesis of Copper-GrapheneNanocomposite Foils with HighHardness

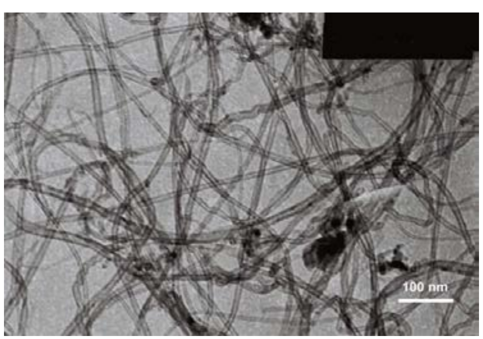
Theories and barriers

4. Cu-CNT (carbon nanotube)





*No new phase is formed, Rule of mix, Volume 45% of CNT (4.4 wt%)**



"High quality" CNT on market

~50%-60% metallic ratio
$50-1000 per gram

*Subramaniam, Chandramouli, et al. "One hundred fold increase in current carrying capacity in a carbon nanotube–copper composite." *Nature communications* 4 (2013).

Theory:

CNT has low or even positive temperature coefficient on electrical conductivity.

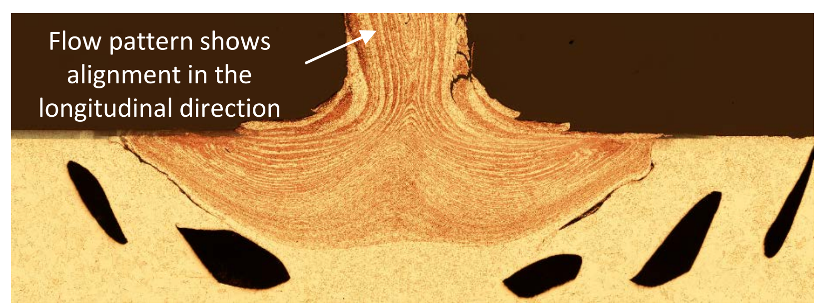
Rule of mixture approach

Barriers:

1. CNT has to be metallic to be conductive.
2. CNT has to be well-aligned.
3. CNT must be long and continuous
4. Good quality control is required even in the lab scale, difficult to scale up.

ShAPE:

Can we use ShAPE to de agglomerate and align the matted CNT? (wires are made, testing)



Electrical conductivity of ShAPE wire is better than competing processes

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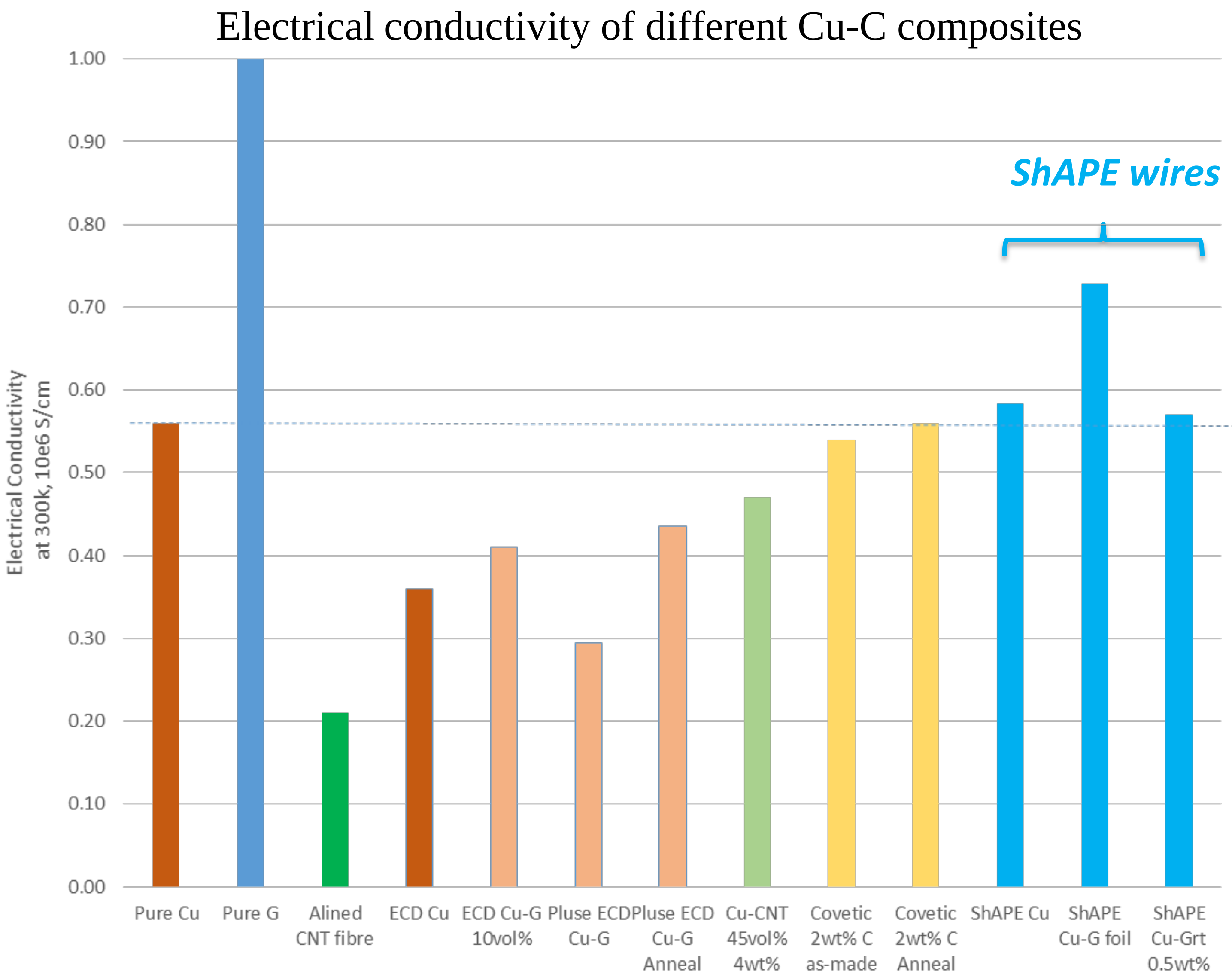

Abbreviations:
G: Graphene,
CNT: carbon nanotube,
Grt: Graphite.
ECD: electrochemical deposition